

**INVESTIGATION OF ELECTROCHEMISTRY  
OF  
HIGH ENERGY COMPOUNDS IN ORGANIC ELECTROLYTES  
BIBLIOGRAPHY ON PROPYLENE CARBONATE,  
 $\gamma$ -BUTYROLACTONE, AND RELATED SUBJECTS  
SUPPLEMENT TO THIRD PROGRESS REPORT  
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by  
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## BIBLIOGRAPHY ON PROPYLENE CARBONATE AND $\gamma$ -BUTYROLACTONE

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## Preface

In the investigation of new high energy electrochemical systems the cyclic esters such as propylene carbonate and  $\gamma$ -butyrolactone have been demonstrated to possess exceptional characteristics. This stems from their chemical, physical, and electrochemical properties. With respect to electrochemical properties, it has been observed that the decomposition voltages for solutions of lithium perchlorate in these solvents are in the 5-6 volt range.

The employment of these solvents in electrochemical systems requires a rather complete knowledge of the chemistry of the compounds. For example, it is intended to use organic depolarizers at cathodes in certain cells. The question arises as to possible condensations, couplings, etc. that these organic depolarizers can undergo with the cyclic esters. Further, it has been observed that aluminium chloride forms highly conducting solutions in these solvents. However, aluminium chloride also catalyzes certain condensations and decompositions of the cyclic esters. It must be known how these catalytic reactions affect the stability of the electrolytic solutions.

Much work has been done and reported in the literature on questions of this type and the purpose of this bibliography is to organize the references in the open literature so that all this previous work will be available. It is expected that this bibliography will furnish a good starting point for future investigations on these solvent systems.

Whereas, the bibliography is fairly extensive, many references in the categories set out below were not included because they did not appear pertinent to the topic of interest, namely, the investigation of the electro-

chemistry of high energy compounds in organic electrolytes. On the other hand, some references were included because they appeared to discuss analogous reactions of interest.

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1. 370-480 mg. in 250 ml. of water.

2. Take 10 ml. and dilute to 100 ml. with water.

3. Take 5 ml. and add 10 ml. hydroxylamine-hydrochloride/sodium hydroxide reagent (25 ml. 3N hydroxylamine-hydrochloride with two drops indicator containing 100 mg. each of phenolphthalein and thymolphthalein in 100 ml. 95% ethanol, 3N sodium hydroxide added to turn purple, then 10-20 drops of indicator added).

4. Heat 20 min. at 40°, cool and shake with 10 ml. ferric allum reagent (25 g. alum in 125 ml. 3.5N nitric acid up to 250 ml. with water), then let stand 10 min.

5. Absorbancy at 505 millimicrons.

% by weight = (11093 A + 77.5)/mg. sample.

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Synthesis.

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Synthesis.

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VII. Evidence for the  $\beta$ -oxidation of omega-fluoro carboxylic acids in vivo.  
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Rambaud, R. and Ducher, S.

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Rothe, J. and Zimmer, H.

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Compt. rend. 227, 437-9 (1948). C. A. 43, 3396-7 (1949).

Preparation of  $\beta$ ,  $\beta$ -diphenyl- $\gamma$ -butyrolactones.

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$\gamma$ -Alkylbutyrolactones.

Japan 26, 417-('65), Nov. 16, Appl. July 27, 1963; 2 pp. C. A. 64, 9600 (1966).

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ortho-phenol fatty acids and their derivatives.

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Schuster, C.

$\alpha$ -Amino- $\gamma$ -butyrolactones.

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$\alpha$ -Amino- $\gamma$ -butyrolactones.

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Synthesis of a  $\beta$ -substituted  $\gamma$ -butyrolactone.

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Isolation of a substituted butyrolactone.

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Studies of  $\gamma$ -butyrolactone derivatives. I. Synthesis of  $\alpha$ -amino- $\gamma$ -butyrolactone.  
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Synthesis via the Friedel-Crafts reaction.

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Synthetic anthelmintics. II.  $\gamma$ -Substituted butyrolactones.  
J. Univ. Bombay 10, Pt. 3, 99-101 (1941). C. A. 36, 3801 (1942).  
Synthesis.

Trivedi, J. J. and Nargund, K. S.  
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J. Univ. Bombay 11, Pt. 3, 127-30 (1942). C. A. 37, 2005-6 (1943).  
Preparation.

Van Tamelen, E. E. and Bach, S. R.  
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J. Am. Chem. Soc. 77, 4683-4 (1955). C. A. 50, 6381 (1956).  
Synthesis.

Vyas, V. A., Bokil, K. V., and Nargund, K. S.  
Synthetic anthelmintics. I.  $\alpha$ -Substituted- $\gamma$ -butyrolactones.  
J. Univ. Bombay 9, Pt. 3, 145-9 (1940). C. A. 35, 6574-5 (1941).  
Synthesis.

Wachs, H.  
Synthesis of a new lactone.  
Riechstoffind 7, 196-7 (1932). C. A. 27, 2685 (1933).  
Preparation of  $\gamma$ -tolyl- $\gamma$ -butyrolactone.

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Holly, F. W., and Folkers, K.  
Synthesis of DL-dimethylidihydro- $\alpha$ -lipoic acid.  
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U. S. 2,421,729, June 3, 1947. C. A. 41, 5550 (1947).

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Weston, A. W.

Amino alkyl phenyl lactones.

U. S. 2,409,937, Dec. 13, 1949. C. A. 44, 2516 (1950).

Synthesis of some  $\alpha$ ,  $\alpha$ -disubstituted- $\gamma$ -butyrolactones.

Zimmer, H., Rothe, J., and Holbert, J. M.

Substituted  $\gamma$ -lactones. V. Synthesis of certain  $\alpha$ ,  $\beta$ -disubstituted  $\gamma$ -lactones. Route to lignans of the  $\alpha$ ,  $\beta$ -dibenzylbutyrolactone class.

J. Org. Chem. 25, 1234-5 (1960). C. A. 55, 5409, 2560 (1961). Makes use of sulfuric acid.

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Berti, F. A.

Amino derivatives of  $\gamma$ -butyrolactone.

Gazz. chim. ital. 84, 420-7 (1954). C. A. 49, 8120-1 (1955).

Bischoff, G.

$\alpha$ ,  $\gamma$ -Dibromobutyryl bromide.

Swiss. 260,302, July 1, 1949 (Cl. 360). C. A. 44, 2549 (1950).

Butyrolactone + red phosphorus + Br<sub>2</sub>  $\xrightarrow{20-60^\circ}$  Title product.

Bischoff, G.

$\alpha$ -Bromobutyrolactone.

Swiss. 264,598, Jan. 16, 1950 (Cl. 360). C. A. 45, 1622 (1951).

At 160-170° for 8 hours followed by cooling and distillation under reduced pressure.

Blicke, F. F. and Brown, B. A.

Interaction of an Ivanov and an Ivanov-like reagent with  $\gamma$ -butyrolactone and  $\gamma$ -valerolactone.

J. Org. Chem. 26, 3685-91 (1961). C. A. 56, 5909-11 (1962).

Reactions.

Dashunin, V. M., Maeva, R. V., Kazaletova, G. A., and Belov, V. N. Substituted lactones and their transformations. III. Hydrogenation of the aromatic nucleus in  $\alpha$ -alkylidenebutyrolactones.

Zhur. Obshchey Khim. 34 (9), 3096-3101 (1964). C. A. 61, 15984-5 (1964). C. A. 58, 470 (1963).

$\gamma$ -Butyrolactone reacts with aldehydes to yield  $\alpha$ -alkyl derivatives.

Dashunin, V. M., Samotuga, G. A., and Belov, V. N.

Substituted lactones and their transformations. V. Hydroxy and oxo derivatives of  $\alpha$ -cyclohexylmethylbutyrolactone.

Probl. Organ. Sinteza, Akad. Nauk. S. S. S. R., Otd. Obshchey i Tekhn. Khim. 1965, 64-8 (Russian). C. A. 64, 6513 (1966).

C. A. 63, 17980 (1965).

Ebel, F. and Weissbarth, O.

$\alpha$ -Acyl lactones.

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$\gamma$ -Butyrolactone reacts with esters in the presence of sodium metal to yield  $\alpha$ -acyl derivatives.

Henry, L.

On butyrolactones and unsymmetrical dimethyltetramethyleneglycol.

Compt. rend. 143, 1221-25. C. A. 1, 714-15 (1907).

$\gamma$ -Butyrolactone reacts with the appropriate Grignard reagent to yield  $\alpha$ ,  $\alpha$ -dimethyltetrahydrofuran.

Isham, R. M.

Succinic and maleic acids from butyrolactones.

U. S. 2,385,518, Sept. 25, 1945. C. A. 40, 1173 (1946).

Oxidation of butyrolactone in the vapor phase with  $V_2O_5$  or  $CuO$ .

Isham, R. M.

Electrolytic oxidation of butyrolactone to succinic acid.

U. S. 2,420,954, May 20, 1947. C. A. 4727 (1947). C. A. 40, 1173 (1946).

Kadyrov, Ch. Sh.

Synthesis of arylbutyric acids. I. Reaction of benzene and chlorobenzene with butyrolactone.

Uzbeksk. Khim. Zhur. 8 (2), 52-7 (1964) (Russian). C. A. 61, 4254 (1964).

$\gamma$ -Butyrolactone reacts with benzene in the presence of aluminum chloride to yield  $\beta$ -phenylbutyric acid.

Knobler, Y., Livergand, S., and Frankel, M.

Preparation of O-phenyl-DL-homoserine and of DL-homoserine from  $\alpha$ -phthalimido- $\gamma$ -butyrolactone.

J. Org. Chem. 24, 1794-5 (1959). C. A. 55, 13325 (1961).

Uses sodium metal, phenol, and  $\gamma$ -butyrolactone.

Korte, F. and Machleidt, H.

Syntheses in the pyran series. I. Rearrangement of  $\alpha$ -hydroxy-methylene- $\delta$ -lactones to 2-methoxy-3-tetrahydropyran carboxylic acid esters.

Chem. Ber. 88, 136-43 (1955). C. A. 50, 5615-16 (1956). C. A. 49, 12453 (1955).

$\gamma$ -Butyrolactone and ethyl formate and sodium in ether yield  $\alpha$ -hydroxymethyl- $\gamma$ -butyrolactone.

Krzikalla, H. and Dornheim, O.

Esters of etherified aliphatic hydroxy carboxylic acids.

Ger. 745,312, Dec. 2, 1943 (Cl. 120, 11). C. A. 40, 3130 (1946).

Synthesis from butyrolactone in the presence of acids.

Krzikalla, H., Plieninger, H., and Maier, K.

Lactone-like products.

Ger. 844,292, July 17, 1952 (Cl. 120, 11). C. A. 52, 10199 (1958).

Synthesis of benzylidene derivatives of  $\gamma$ -butyrolactone.

Mayhew, R. L. and Williams, E. P.

Nematicidal  $\alpha$ -halogenated- $\gamma$ -butyrolactones.

U. S. 2,974,084, March 7, 1961. C. A. 55, 14807 (1961).

Michael, A. and Weiner, N.

Formation of enolates from lactonic esters.

J. Am. Chem. Soc. 58, 999-1005 (1936). C. A. 30, 5183-4 (1936).  
 $\gamma$ -Butyrolactone does not react with sodamide in diethyl ether.  
It reacts with pulverized sodium or with 40% sodium amalgam only slightly upon long standing. No experimental data is given to substantiate this statement.

Oelshlaeger, H., Schmersahl, P., and Toporski, W.  
Synthesis of new local anesthetics. IV.  $\gamma$ -Halobutyric acids from  $\gamma$ -butyrolactone and several new omega-amino-3-alkyl-6-haloacrylanalides.

Arch. Pharmacol. 294, 488-98 (1961). C. A. 56, 3342-3 (1962).  
C. A. 55, 19835 (1961). C. A. 53, 3136 (1959).

$\gamma$ -Butyrolactone +  $\text{SOCl}_2$  =  $\gamma$ -chlorobutyryl chloride.

$\gamma$ -Butyrolactone +  $\text{HCl}$  =  $\gamma$ -chlorobutyric acid.

$\gamma$ -Butyrolactone +  $\text{HBr}$  =  $\gamma$ -bromobutyric acid.

$\gamma$ -Butyrolactone +  $\text{HCO}_2\text{H}$  = pentanedioic acid.

$\gamma$ -Butyrolactone +  $\text{HI}$  with  $\text{P}_2\text{O}_5$  =  $\gamma$ -iodobutyric acid.

Petrov, G. I.

Free radical addition of butyrolactone and unsaturated compounds.  
Izvest. Akad. Nauk S. S. R., Otd. Khim. Nauk. 1962, 146-51.  
C. A. 57, 16390 (1962). C. A. 56, 308, 7155 (1962). C. A. 55, 22093 (1961).

Plieninger, H.

Cleavage of  $\gamma$ -butyrolactone and  $\alpha$ -amino- $\gamma$ -butyrolactone with sodium methyl mercaptide or selenide. A synthesis of methionine.  
Chem. Ber. 83, 265-8 (1950). C. A. 44, 9919 (1950).

$\gamma$ -Butyrolactone +  $\text{RSNa} \rightarrow \text{RS}(\text{CH}_2)_3\text{COOH}$ .

Rambaud, R., Ducher, S., and Boudet, R.

Butenolide. III.

Bull. soc. chim. France 1956, 1419-24. C. A. 51, 3602 (1957).  
C. A. 25, 4850 (1931).

Reactions.

Schotte, L.

$\gamma$ -Butyrolactone reactions involving ring fission.  
Arkiv. Kemi 8, 457-61 (1955). C. A. 51, 1838 (1957).

Spath, E. and Lintner, J.

Formation of lactams from lactones.

Ber. deut. chem. Ges. 69B, 2727-31 (1936). C. A. 31, 2172 (1937).  
 $\gamma$ -Butyrolactone to  $\gamma$ -butyrolactam.

Stacey, M., Barker, S. A., Ward, R. B., Grant, P. M., and Lloyd, I. R. L.

Polyamino and polyhydroxycarboxylic acids.

Brit. 901,037, July 11, 1962, Appl. Jan. 7, 1959; 8 pp. C. A. 58, 3519 (1963).

Polymerization of  $\gamma$ -butyrolactone by irradiation.

Stepanov, F. N.

3-indolebutyric acid.

U. S. S. R. 66,681, July 31, 1946. C. A. 41, 2087 (1947).

Suzuki, K.

Synthesis of resorcylobutyrolactone mono- and dimethyl esters.  
Bull. Inst. Phys. Chem. Research (Tokyo) 15, 71 (1936). C. A. 31, 6640 (1937).

Treatment of the acid form with sodium amalgam and dilute acetic acid results in formation of the lactone.

Takayama, Y. and Mizuno, S.

Formation of succinic acid from tetrahydrofurfuryl alcohol and  $\gamma$ -butyrolactone by electrolysis.  
J. Electrochem. Soc. Japan 18, 298-301 (1950). C. A. 45, 8923 (1951).

Talbot, G., Gauaby, R., and Berlinguet, L.

Synthesis of 4-aminobutyric acid and 2, 4-diaminobutyric acid from butyrolactone.

Can. J. Chem. 36, 593-6 (1958). C. A. 52, 15425-6 (1958).

Tani, H. and Fudo, K.

Reaction of  $\gamma$ -butyrolactone with alkyl mercaptans.

Mem. Inst. Sci. Ind. Res., Osaka Univ. 6, 100-1 (1948). C. A. 42, 10198 (1951).

Wagner, A. F.

Substituted butyric acids.

U. S. 2,842,587, July 8, 1958. C. A. 52, 18221 (1958).

$\gamma$ -Butyrolactone reacts with  $PBr_3$  at  $90-135^\circ$  to yield  $\alpha$ ,  $\gamma$ -dibromo-butyric acid.

Wagner, A. F.

Addition product of metal hydrocarbon mercaptide with an  $\alpha$ -halo- $\gamma$ -butyrolactone.

U. S. 2,872,458, Feb. 3, 1959. C. A. 53, 12184 (1959).

Yur'ev, Yu. K., Vendel'shtein, E. G., and Zinov'eva, L. A.

Pyrrole derivatives. XXXV. Transformation of butyrolactone into 2-thiophanone, 2-pyrrolidone, and 1-phenyl-2-pyrrolidone.

Zhur. Obshchei Khim. (J. Gen. Chem.) 22, 509-13 (1952). C. A. 47, 2747-8 (1953). C. A. 46, 8086 (1952). C. A. 44, 5869 (1950).

Zimmer, H. W. and Holbert, J. M.

Butyrolactone derivatives.

U. S. 3,030,361, April 17, 1962, Appl. July 14, 1958; 5 pp. C. A. 57, 9742-3 (1962).

Zimmer, H. W. and Walter, R.

Substituted  $\gamma$ -lactone. XX. Reaction of  $\alpha$ -benzylidene- $\gamma$ -butyrolactones with bromine and some chemical properties of the resulting products.

J. Heterocyclic Chem. 2 (4), 477-8 (1965) (English). C. A. 64, 9631 (1966). C. A. 63, 1728 (1965). C. A. 63, 4236 (1965).

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Bremer, K. H. and Seidel, L.  
The enolization of  $\gamma$ -lactones.  
Angew. Chem. 76 (9), 376 (1964). C. A. 61, 1751-2 (1964).  
Grignards.

Bryusova, L. Ya., Simanovskaya, E., and Ul'yanova, A.  
Substituted butyrolactones and their transformation into cyclopentanone derivatives.  
Sintez Dushistykh Veshchestv, Sbornik Statei 1939, 165-77;  
Khim. Referat. Zhur. 1940, No. 4, 115. C. A. 36, 3784 (1942).  
Synthesis.

Curtius, T. and Sauerberg, H.  
Action of hydrazine upon butyrolactone- $\alpha$ -carboxylic esters.  
J. prakt. chem. 125, 139-51 (1930). C. A. 24, 3215 (1930).  
Carbethoxybutyrolactone + hydrazine + water = ethyl  $\beta$ -hydroxyethylmalonyl hydrazine. Carbethoxybutyrolactone + an excess of hydrazine + water = the dihydrazide. Carbethoxybutyrolactone + nitrous acid = butyrolactone- $\alpha$ -carboxylic azide. Carbethoxybutyrolactone + para-methylaniline =  $\beta$ -hydroxyethyl-malonyl di-para-toluidide.

Feofilaktov, V. V. and Onishchenko, A. S.  
Action of nitrous acid on  $\alpha$ -substituted butyrolactones. I.  
J. Gen. Chem. (U. S. S. R.) 9, 304-13 (1939). C. A. 34, 378 (1940).  
Action depends on the nature of the  $\alpha$ -substituent.

Feofilaktov, V. V. and Onishchenko, A. S.  
Action of phenyl diazonium chloride on  $\alpha$ -substituted butyrolactones. II.  
J. Gen. Chem. (U. S. S. R.) 9, 314-24 (1939). C. A. 34, 378-9 (1940).  
Similar to the action of nitrous acid but no polymerization or isomerization.

Fischhoff, G.  
Derivatives of  $\alpha$ -keto- $\gamma$ -butyrolactone. Ethyl ester of  $\alpha$ -keto- $\gamma$ -butyrolactone- $\beta$ -carboxylic acid ( $\alpha$ -keto-paraconic ester) and its  $\beta$ -hydroxymethyl derivative.  
Ann. chim. (12), 6, 227-45 (1951). C. A. 45, 9475-78 (1951).

Gault, H.  
Derivatives of  $\alpha$ -keto- $\gamma$ -butyrolactone.  $\gamma$ -Alkyl- and  $\gamma$ -aryl- $\alpha$ -keto- $\gamma$ -butyrolactone- $\beta$ -carboxylic esters. Introduction.  
Ann. chim. (12), 6, 220-6 (1951). C. A. 45, 9475-78 (1951).

Harradence, R. and Lions, F.  
Indoles. VIII. 3-hydroxymethylindole-2-carboxylic lactone.  
J. Proc. Royal Soc. N. S. Wales 72, 221-7 (1939). C. A. 33, 6338-9 (1939).  
Acetobutyrolactone is the starting material.

Kawasaki, H.  
Vitamin B<sub>1</sub> and related compounds. LXXVI. Thioltype thiamine

derivatives. 7. S-acylation of thiazolium compounds.  
J. Pharm. Soc. Japan 76, 702-5 (1956). C. A. 51, 429-30 (1957).  
C. A. 50, 13044 (1956).  
Treat  $\alpha$ -benzoylbutyrolactone with  $\text{SO}_2\text{Cl}_2$  to get  $\alpha$ -chloro derivatives.

Kohles, E. P. and Kimball, R. H.  
Hydroxy derivatives of  $\alpha$ ,  $\gamma$ -diphenylbutyric acid.  
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G. Condensations.

(b)  $\gamma$ -Butyrolactone.

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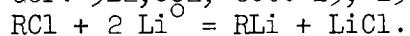
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